

Long-Term Environmental Durability of F/A-18 Composite Material

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DSTO-TR-0826

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**Airframes and Engines Division
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ABSTRACT

The long-term environmental durability of AS4/3501-6 graphite/epoxy composite was evaluated by a ground exposure trial conducted at four locations within Australia and one in Malaysia for a period of between 468 and 520 weeks. The combined effects of moisture uptake, UV degradation, temperature, rainfall and wind on the strength of the samples were all evaluated. The coupons absorbed an average of about 0.9% moisture by weight over the length of the trial. The residual strength of the coupons was determined using the interlaminar shear strength (ILSS) test (ASTM D2344) both at ambient temperature and 100°C. A statistical analysis of the results determined that the combined effect of moisture and elevated temperature (100°C) decreases ILSS by about 10%. A small enhancement in ILSS at room temperature was noted for specimens which absorbed moisture over the period of the trial. This is likely to be due to the combined effects of stress relief and plasticisation of the matrix. No other significant degradation mechanism was identified in this study apart from a slight decrease in ILSS of about 5% for specimens which were coated with the standard F/A-18 paint scheme. This may be attributed to the solvents used in the application of the paint. However, the benefits of the paint protecting the composite surface from erosion and UV degradation far outweigh this structurally insignificant change in ILSS.

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Published by

*DSTO Aeronautical and Maritime Research Laboratory
PO Box 4331
Melbourne Victoria 3001 Australia*

*Telephone: (03) 9626 7000
Fax: (03) 9626 7999
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AR-010-987
June 1999*

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Long-Term Environmental Durability of F/A-18 Composite Material

Executive Summary

Composite materials are used widely in modern fighter aircraft such as the F/A-18. The performance of the composite material, in particular the matrix, is of prime importance for the long-term durability of the aircraft. Environmental factors such as atmospheric humidity, rainfall, UV radiation, wind and temperature can degrade the performance of a composite material over the long term. The use of a real-time long-term exposure trial to determine the effects of these conditions on the performance of the material is of great benefit.

A ground exposure trial was initiated by DSTO-AMRL at a number of exposure sites around Australia as well as Malaysia. Coupons of graphite/epoxy composite material representative of a F/A-18 speed brake were exposed at these locations under three different conditions; fully covered, partly shaded and fully exposed to sunlight. The coupons were also evaluated using two types of paint schemes as well as the unpainted case. After more than eight years of outdoor exposure the coupons were returned to DSTO-AMRL and tested to determine the interlaminar shear strength (ILSS); a test of the composite matrix behaviour under shear loading. These tests were performed both at room temperature and at 100°C. The determination of coupon moisture content was evaluated by drying the specimens at 80°C and these dried samples were subsequently tested to determine the effects of moisture on ILSS. Typically the coupons absorbed between 0.7% - 0.9% moisture by weight.

The complete test program consisted of over 2100 specimens. In order to draw meaning from this large data set and to elucidate the major effects of all the variables considered a statistical analysis was performed. The results showed that the combined effect of moisture and elevated temperature (100°C) decreases ILSS by about 10%. A small enhancement in ILSS was noted for specimens after absorbing moisture compared to when they were re-dried to zero moisture content when tested at room temperature. This is likely to be due to the combined effects of stress relief and plasticisation of the matrix. No other significant degradation mechanism was identified in this study apart from a slight decrease in ILSS of about 5% for specimens which were coated with the standard F/A-18 paint scheme. This may be attributed to the solvents used in the application of the paint. However, the benefits of the paint protecting the composite surface from erosion and UV degradation far outweighs this structurally insignificant change in ILSS.

In conclusion this long-term study showed that ground based environmental exposure does not adversely affect the performance of the F/A-18 composite matrix to any significant degree. Long term trials such as this continue to provide valuable information on the long-term durability of composites and provide confidence in their ability to operate under conditions representative of RAAF service use.

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1. Introduction

Environmental testing of composite materials is an essential part of the certification process for an aerospace component. A number of environmental factors experienced by military aircraft both on the ground and during flight can influence the long-term durability of the material. It is therefore vital to assess any possible degradation which may be occurring in typical service.

Environmental factors for composite materials include humidity, temperature, ultraviolet radiation levels, wind conditions and rainfall. All these factors can combine to degrade the composite material over the long term. This degradation can be quite severe in some cases. The greatest effect on composite materials is typically diffusion of moisture. The epoxy matrix of most composite materials can pick up moisture from humid air which degrades mechanical properties. At elevated temperature reductions in matrix-based mechanical properties can be as high as 25%. Although the effects of moisture can be reasonably well simulated in the laboratory using environmental conditioning chambers in a short period of time, the effects of long-term exposure cannot be predicted reliably. The use of real-time long-term environmental conditioning trials can provide very good indications of a materials performance after many years of service.

The material system chosen for this trial is the same as that used in the F/A-18 aircraft, which has approximately 34% of its external surface constructed from graphite/epoxy composite. Although other countries such as the U.S.A, Canada and Spain also operate this aircraft, Australia has unique climatic conditions, including high levels of humidity in the tropics, low temperatures at high altitude, and intense sunlight.

In this study a number of graphite/epoxy coupons were subjected to a range of exposure conditions, locations and paint coating schemes. The weight-gain of these coupons was monitored over a period in excess of eight years at five locations in Australia and Malaysia. At the conclusion of this trial the coupons were returned to the laboratory and evaluated using the Apparent Interlaminar Shear Strength mechanical test (ASTM D2344). A statistical analysis was then performed on the data (which totals over 2000 tests) to evaluate the extent of any environmental degradation and which conditions produced the greatest effect.

2. Specimen Manufacture

Laminates manufactured from Hercules AS4/3501-6 graphite/epoxy material were laid up to represent construction of a F/A-18 speed-brake [1]. The speed-brake construction consists of a ten ply lay-up ($\pm 45/0/90/0$ s). Since moisture can diffuse through only one face of the composite skin of the F/A-18 speed brake (the honeycomb construction means that only one side of the laminate is exposed to ambient air), coupons were manufactured at twice the thickness in order to remain representative

(ie: $(\pm 45/0/90/0)_{2s}$). Laminates were cured using the following manufacturers recommended cure cycle:

1. Vacuum and 586kPa external pressure
2. Heat to 116°C in 45 minutes and dwell for one hour
3. Vent vacuum and raise pressure to 680kPa
4. Heat to 175°C in 45 minutes and hold for two hours
5. Post-cure at 175°C for four hours.

Coupons were then cut to size 105mm by 45mm. Details are reported in [1].

2.1 Paint Schemes

Two thirds of all the specimens were painted using two types of paint schemes (A and B), while the remainder were left unpainted. The paint used was the same as that applied to the F/A-18 aircraft and consisted of a two-part epoxy polyamide anti-corrosive primer and a two-part aliphatic polyurethane enamel finishing coat. Solvents used in the paint include xylene, methyl ethyl ketone and cellosolve acetate [2].

3. Exposure Details

The exposure trial used here is designed to simulate the effects of ground exposure only. Work conducted by NASA [3] found that ground exposure produced higher levels of moisture uptake compared to coupons placed on aircraft. Ground exposure is therefore a more severe evaluation of the 'wet' condition. Ground exposure does not however simulate extreme high temperature excursions which can be experienced during supersonic flight, the influence of service stresses or damage to the composite matrix through thermal spiking [4].

3.1 Exposure Locations

A number of geographical locations both around Australia and Malaysia were chosen in order to simulate the effects of environment at all locations relevant to routine RAAF service. These sites (Table 1) have varied humidity, temperature, rainfall and ultraviolet exposure conditions.

Table 1 List of exposure locations for long-term exposure trial

<u>Exposure Site</u>
Tindal, Northern Territory
AMRL-Q, Innisfail, Queensland
Williamtown, New South Wales
Darwin, Northern Territory
Butterworth, Malaysia

Butterworth, Darwin and AMRL-Q are all tropical locations. Tindal represents a tropical environment but is further inland from the coast than Darwin and experiences a prolonged dry season. Williamtown represents a sub-tropical coastal climate (Latitude 32.8° South).

3.2 Exposure of specimen coupons

Specimens were placed in a stainless steel fixture in such a way that all specimens experience the prevailing levels of humidity but experience three different degrees of exposure to solar radiation. Specimens classed as "exposed" are fully exposed to solar radiation during the day, "protected" specimens are fully protected from direct incident radiation by a stainless steel plate, while "shaded" specimens are placed beneath a piece of aluminium honeycomb which restricts direct exposure to radiation to only a few hours at midday. Figure 1 shows a photograph of a typical exposure cabinet. The specimens were progressively recalled over a period of twelve months giving total exposure times between 468 and 520 weeks.



Figure 1: Coupon exposure cabinet

4. Moisture Uptake

The uptake of moisture in the coupons was tracked regularly using the coupon weight and plotted over time. Four coupons of each type were weighed regularly and the results averaged. A stainless steel calibration weight ensured consistency and accuracy of weighing between the different exposure sites over the long term of the trial.

A typical plot is shown in Figure 2. This shows the moisture uptake for specimens in the exposed, shaded and protected state. Note that the protected coupons have the highest moisture content as they are not subject to direct sunlight which dries the composite. Surface temperatures due to solar radiation can be up to 85°C for the paint scheme considered. Also note that decreases in weight occur due to loss of epoxy material from the exposed surface layer due to ultraviolet degradation.

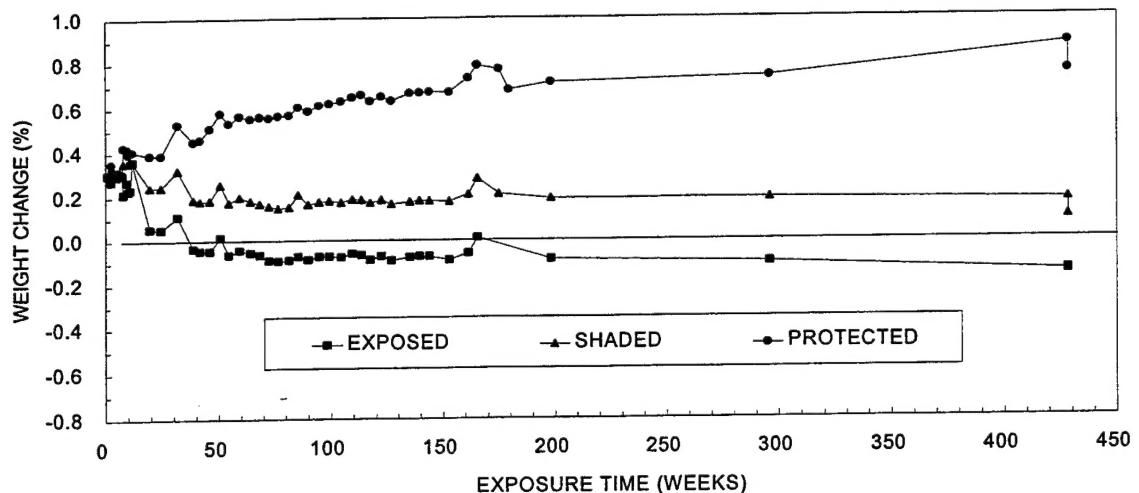


Figure 2: Weight changes for unpainted coupons trialed at Butterworth

Details of the moisture absorption behaviour of these same coupons has been reported earlier [5]. Table 2 shows the level of moisture absorbed by the protected coupons as measured by drying the coupons at 80°C until a constant weight was achieved.

Table 2: Equilibrium moisture content(w/w %) of composite coupons from [5]

	Exposure Site				
	Butterworth	Darwin	Tindal	Williamtown	AMRL-Q
Equilibrium Moisture Level	0.94%	0.88%	0.66%	0.80%	0.90%

Note that the tropical locations show a similar high value of moisture content (around 0.9%) while Tindal shows a lower value due to its severe dry season condition.

5. Apparent Interlaminar Shear Strength Testing

Interlaminar shear strength (ILSS) testing was performed using ASTM D2344. The main difference between the ILSS test performed here and those regularly used is that the layup used here is multi-directional. This does not allow the results to be compared directly with manufacturer's data which is for unidirectional layups.

Tests were performed for both the as-received state ('wet') and after drying (at 80°C) to remove all moisture ('dry'). Tests were also performed at two temperatures, ambient and 100°C. The number of tests totalled in excess of 2000 which required careful management of both the specimens and the test data.

5.1 Specimen Preparation and Test Procedure

Coupons were weighed on arrival and then cut into ILSS specimens 10mm by 12mm using a diamond saw. A central core of 20mm by 100mm was also cut out from each moisture coupon and dried to determine the moisture content.

5.1.1 Elevated Temperature Tests

An insulated metal chamber with removable doors was used as an oven. Heating of the chamber was achieved by using an industrial hot air gun and the temperature was monitored using a thermocouple placed close to the specimen.

5.1.2 Elevated Temperature 'Wet' Tests

Steam was used for elevated temperature wet tests in order to reduce the possibility of specimen drying. Each ILSS test lasts for about ten minutes and it was found that no loss in weight was experienced if steam was introduced in the specimen chamber. The steam was generated by a commercially available hot water urn. A copper cooling coil supplied by compressed air was used to keep the load cell cool. The setup is illustrated in Figure 3.

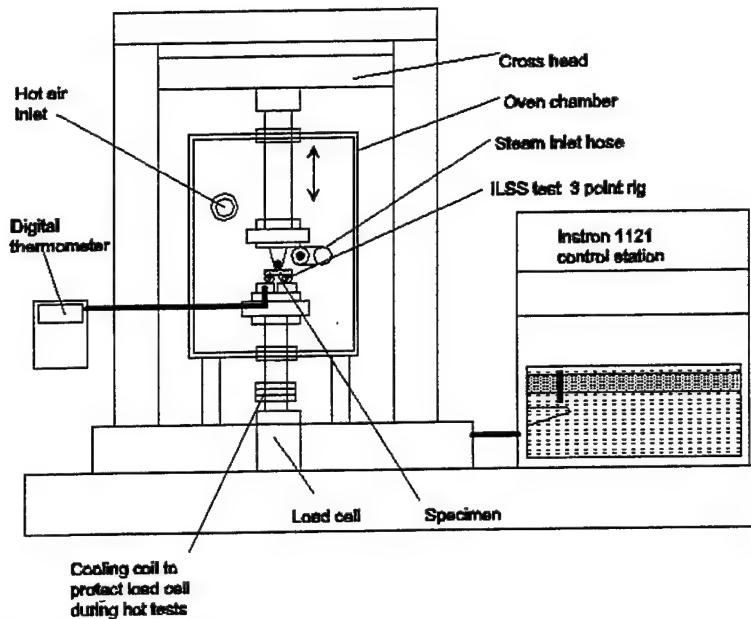


Figure 3: Test setup for ILSS testing

5.1.3 Specimen storage and drying

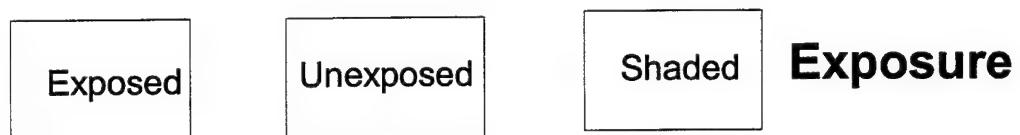
Specimens were marked to be tested either in the as-received state and after complete drying at 80°C. Specimens marked for dry tests (re-dried after exposure) were dried until constant mass in a central core cut-out from a moisture coupon was achieved after a number of consecutive days.

To prevent problems in specimen identification, as well as allowing the circulation of air within the oven, all specimens and their respective central core cut-out sections were placed in wire mesh envelopes with a metal identification tag. Also, to prevent mix up of specimens between geographical locations (Table 1), each location was allocated a separate shelf in the oven and only one was tested at a time.

5.2 Summary of Test Matrix

Figure 4 shows a chart summarising the full test matrix and the variables investigated.

Location



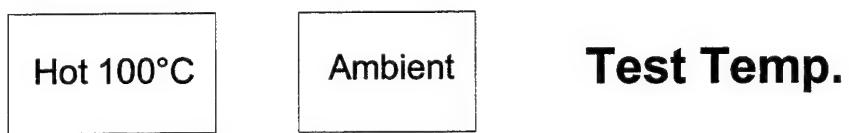
Exposure



Paint



Moisture



Test Temp.

Figure 4: Summary of ILSS test matrix variables

6. Statistical Analysis

Due to the large number of specimens and variables involved in this program it was decided to undertake a thorough statistical analysis of the results to elucidate the major effects on ILSS strength after long-term environmental exposure.

6.1 Approach to the problem

The aim of the statistical analysis was to determine whether there were any statistically significant differences in ILSS as a result of either moisture content, test temperature, paint scheme, degree of exposure or location. In order to conduct the statistical analysis the ILSS values were copied into a Microsoft Excel spreadsheet and assigned codes to designate the different conditions. A data analysis workbook, XL Statistics [6] was then used to do the analysis. This program provided basic statistical information such as the mean, standard deviation, skew and possible outliers.

As there are 180 different combinations of these variables there was no simple approach to comparing them. While there were over 2100 specimens tested, each different combination had at most twelve ILSS values to compare. A power analysis using XLStatistics indicated that a minimum of 30 values were required if a comparison was to be made with any statistical confidence.

It was therefore decided to group data in order to produce analyses with good confidence. The approach taken was to first compare those variables that would have had the largest effect on the results and whose effects are well understood. Therefore the first comparison was between the four different combinations of moisture content and test temperature. The difference between these four combinations was deemed statistically significant and therefore would have to remain separated for comparisons of the other variables. Therefore in order to compare the effects of paint scheme the data was further broken down into the twelve different combinations of moisture content, test temperature and paint scheme [Table 4]. After comparing these it was then possible to further break down the data to compare the effects of the three different types of exposure, while still maintaining large enough samples to ensure statistical confidence. As mentioned before it was not possible to break down the data further to compare location as the sample size would become too small. Therefore, in order to compare the effects of location, the data was broken down according to moisture content, test temperature and location.

6.2 Statistical method

Before any comparison could be made, the mean, standard deviation and skew were calculated and any outliers removed. In order to compare the mean values of two sample populations it had to be shown that any difference between the two was statistically significant and could not be attributed to chance. In order to do this hypothesis testing was used which aims to 'disprove' a null hypothesis, H_0 in favour of

an alternative hypothesis, H_a . For comparison of mean values (μ), the null and alternative hypotheses are:

$$H_0: \mu_1 - \mu_2 = 0$$

$$H_a: \mu_1 - \mu_2 \neq 0$$

The null hypothesis expression suggests that samples one and two are actually from the same population. The alternative hypothesis suggests that samples one and two are not from the same population, if this is the case it can then be tested if μ_1 is greater than or less than μ_2 . For this the XLStatistics 2-sample t-test was used. The following assumptions were verified before reporting results [6]:

1. The samples are simple random samples (every sample of the same size is equally likely to be chosen) of independent observations (the result of one measurement doesn't affect the result of others) drawn from the two populations.
2. Either the populations are approximately normally distributed or the sample sizes are sufficiently large ($n \geq$ about 30 is a good rule of thumb). A plot of residuals or a normal probability plot can be useful for checking normality.

For comparisons of the ILSS values, from two different groups, the one number, two group (1num2grp.xls) worksheet was used. This worksheet provides a number of basic tests of the data and provided a suitable hypothesis test. XL Statistics provides a p-value (probability) which if less than 0.05 (the significance level) suggested that the probability of the null hypothesis being true was less than 5% and therefore the null hypothesis would be rejected in favour of the alternative hypothesis. For example, the two groups being compared may have been the ILSS values for unexposed and exposed specimens. If the p-value had been less than 0.05 it would suggest that there was a statistically significant difference between the mean ILSS values for exposed and unexposed composites.

6.3 Results of analysis

After completing the comparisons and calculations of p-values the results were analysed for any consistent trends. It was found that the conditions that had a statistically significant effect were moisture content and test temperature, which was expected. It was also found that there was a significant difference between the painted and unpainted specimens, however, there was no significant difference between the two paint schemes, A or B. Unpainted specimens had consistently greater ILSS values. When comparing the effects of exposure and location there were some statistically significant differences, however, there were no consistent trends and therefore it was decided that location and exposure type have no consistent effect on the ILSS values of composites. The results for comparing moisture content and test temperature are shown in Table 3, and the results for effects of paint are shown in Table 4.

Table 3: Values of ILSS for moisture and temperature effects for all results

Sample / Test	Mean ILSS (MPa)	Standard Deviation (MPa)
As received/room temp	72.9	8.3
As received/hot (100°C)	54.7	6.2
Dried/room temp.	62.3	7.1
Dried/hot (100°C)	55.7	9.5

Table 4: Effects of paint on ILSS - unpainted coupons (shaded) show consistently higher ILSS values for all conditions. No difference in ILSS is seen for the paint type (A or B).

Specimen Designation	Mean ILSS (MPa)	Standard Deviation (MPa)
AHA	53.6	5.7
AHB	53.7	5.0
AHU	57.0	7.3
ARA	72.4	7.2
ARB	71.1	8.9
ARU	75.2	8.3
DHA	54.6	9.4
DHB	54.5	8.4
DHU	58.1	10.4
DRA	61.5	7.4
DRB	60.6	6.9
DRU	64.8	6.4

First letter: A = as received, D = dried
= 100°C test

Second letter: R= room temperature test, H

Last Digit: Paint Scheme A, B or Unpainted.

7. Failure Analysis of ILSS Coupons

Failure analysis of the ILSS coupons was performed using an OLYMPUS SZH-10 microscope at magnifications up to 64X. This allowed the interlaminar cracks to be readily observed.

The failure modes observed here are inter-laminar, however, the fracture did not occur at the centre of the specimen at the $0^\circ/0^\circ$ interface of the $[+45,-45,0,90,0]_{2s}$ layup. In all of the specimens the cracks initiated at the edges of the $0^\circ/90^\circ$ interface. Some specimens had twin cracks that initiated from opposite $0^\circ/90^\circ$ interfaces whereas others had only a single crack. Crack propagation varied between the specimens, with some cracks continuing along the $0^\circ/90^\circ$ interface and others crossing over plies. This change in crack direction would most likely have been caused by defects in the composite. Schematics of typical failure modes appear in Figure 5 and Figure 6. Most specimens failed in this way however in some cases the crack moved to a different ply/ply interface during failure.

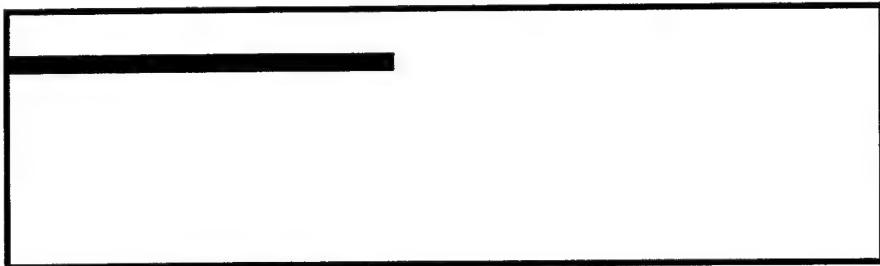


Figure 5: Single crack at $0^\circ/90^\circ$ ply interface

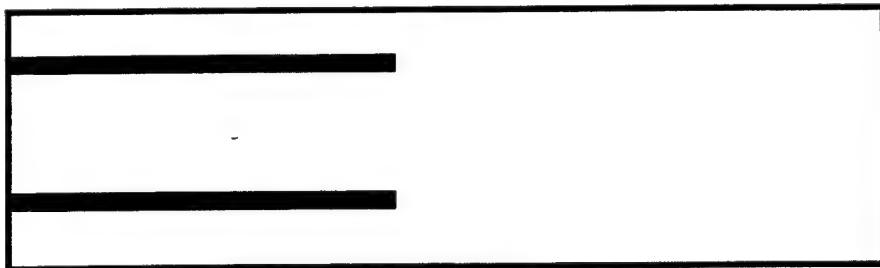


Figure 6: Double crack at $0^\circ/90^\circ$ ply interfaces

This type of failure mode also had a noticeable effect on the ILSS strength compared to a unidirectional layup. Research by Harding et al. [7] showed that quasi-static ILSS tests on graphite/epoxy specimens for $0^\circ/90^\circ$ and $+45^\circ/-45^\circ$ layups were approximately 35% lower than for a unidirectional ($0^\circ/0^\circ$) layup. The ILSS values seen in this trial are consistent with the results demonstrated by Harding et al. Baseline ILSS values for this trial were 64.2 MPa compared with the manufacturer's data of 124 MPa for a unidirectional composite. Reasoning for lower ILSS values for a multi-directional layup was given by Harding et al. as [7] 'to the closer packing possible between adjacent plies when the fibre directions are aligned rather than orthogonal, leading to a better bonding between the plies'.

8. Further Testing

Further testing was carried out in order to resolve some outstanding issues apparent from the statistical analysis. This concerns the lower ILSS of painted specimens and the determination of baseline ILSS values.

8.1 Effect of Paint on ILSS

Since the ILSS values for unpainted specimens were consistently greater than those which were painted with paint schemes A or B it was decided to investigate this effect. Previous reports [5] also found that painted specimens did not have differing moisture contents compared to the unpainted specimens so this could not be a factor. The effects of solvents in the paint during application was considered a possibility and evaluated using ILSS tests on unpainted coupons which had been kept for the same time as the exposure trial in laboratory air.

To test the effects of solvents three sets of ILSS specimens were cut from the coupons. One was soaked in a mixture of acetone and Methyl Ethyl Ketone (MEK) for one day, the second was soaked in a mixture of xylene and toluene for one day, and the third was kept as a control. ILSS tests were conducted and the results are shown in Table 5.

Table 5: Effects of solvents on ILSS

Solvent exposure	Mean ILSS (MPa)	Standard Deviation (MPa)
Baseline	68.7	3.4
Acetone/MEK	52.2	1.3
Xylene/Toluene	55.1	7.7

This shows that lightweight volatile solvents such as acetone and MEK can have a noticeable effect on ILSS. Although this test does not fully prove the loss of ILSS for the painted coupons it is however a strong possibility.

8.2 Determination of baseline ILSS

The baseline ILSS values were obtained for both the as-manufactured state and after exposure in a laboratory environment for the same period of time as the exposure trial. Coupons which were kept in a laboratory environment were tested both in the original condition and after being dried at 90°C. The drying curve for four coupons is shown in Figure 7 which shows that laboratory air provides enough humidity to produce a moisture level of about 0.64% (w/w) in the composite.

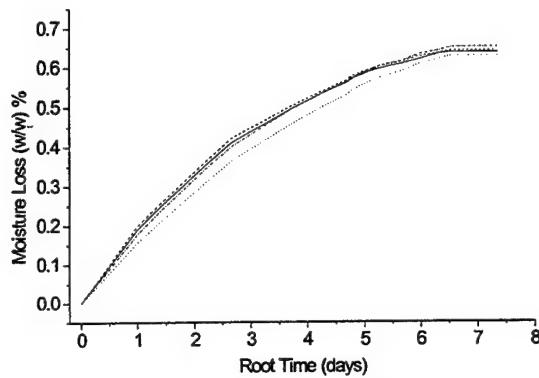


Figure 7: Moisture loss curves for laboratory stored specimens (drying at 90°C)

Coupons were cut to 10mm by 12mm and tested for ILSS at both ambient and elevated (100°C) temperature. Baseline results shown in Table 6 compare closely with those gained from the long-term trial (Table 3).

Table 6: Baseline ILSS results for specimens stored in laboratory over length of trial

Test Type	ILSS (mean) MPa	ILSS (standard deviation) MPa
As-manufactured - room temp.	64.2	2.5
Laboratory conditioned - room temp.	68.7	3.4
Laboratory conditioned - hot (100°C)	54.9	0.6
Dried - room temp.	59.8	5.3
Dried - hot (100°C)	52.2	5.4

9. Discussion

The results broadly show that at room temperature the wet (as received specimens) had greater ILSS than after drying to zero moisture content, while at elevated temperature (100°C) the dry specimens have greater ILSS than those wet (as received). Also the painted specimens were lower in ILSS than the unpainted ones. The type of exposure and exposure location had no consistent effect on ILSS.

It appears that the presence of moisture within the matrix at temperatures well below the glass transition temperature (T_g) actually provides an increase in ILSS (about 15%), whereas at elevated temperatures, the presence of moisture reduces the ILSS by about 10%. A similar behaviour has been noted by Joshi et al. [8] with the testing of $\pm 45^\circ$ graphite/epoxy composites. Joshi attributed the increase of strength at low temperatures to the moisture providing a release of residual strains induced by differential thermal contraction during cooling. This theory may not necessarily apply as the ILSS test produces an unstable failure that initiates from some flaw in the specimen. Therefore it may also be likely that moisture in the matrix, at temperatures well below T_g , provides an element of fracture toughness to the matrix that delays the initiation and propagation of an inter-laminar crack. At higher temperatures the added moisture would lower the T_g therefore resulting in a more plastic behaviour and a lower ILSS value than the dry specimens.

The results also suggest that painting the specimen, which protects it from UV degradation, reduces the inter-laminar shear strength by a small amount (5%). The first possibility was that the paint may have been trapping moisture causing a larger percentage of water absorption. However, the drop in ILSS values was independent of moisture content and test temperature, therefore, this possibility was discarded. The second possibility was that the paint was providing an added measured thickness to the specimen that was not load bearing which would result in a lower value for the ILSS. However the difference in thickness was less than 1% on average whereas the difference in ILSS was in the order of 5%. As it still appeared that something else in the paint may be affecting the composite, a request was sent for information regarding the chemicals found in the paint. The effects of paint solvents used in their application was considered as a possibility. Solvents commonly used in these paints were gathered by [2] and tests were performed to assess their effects (Section 8.1). These results showed a drop of approximately 20 to 24% in the ILSS properties as a result of a 24 hour soak in the solvents. Losses of up to 80% in ILSS strength have been recorded after 30 day soaks in acetone [9]. Although this result is inconclusive and the decreases in ILSS found are not as severe as found in these tests it may warrant further investigation at a later stage.

Comparison of baseline ILSS values (Table 6) with those from the long-term trial (Table 3) show that the environmental exposure has had no significant effect on matrix properties.

10. Conclusions

A long-term ground exposure environmental trial was conducted at four locations around Australia and one in Malaysia. After over eight years of exposure under a variety of conditions the specimen coupons were recalled and tested using the interlaminar shear strength test (ILSS). A statistical analysis was then performed to determine which factors produced the most significant effects on material properties. The greatest effect was that of absorbed moisture from humid air which decreased ILSS

by about 10% at elevated temperature (100°C). The exposure site and other factors such as exposure to direct solar radiation did not produce significant property changes. A small effect of decreasing ILSS (about 5%) was noted for specimens which were painted with the standard F/A-18 paint scheme. This is possibly due to the solvents used in the paints affecting the composite matrix. However the benefits of painting to protect the composite surface from erosion and UV degradation far outweigh this insignificant change in ILSS.

11. Bibliography

- Statistics - A First Course, J.E. Freund, Prentice-Hall, Inc., 1976.
- Mathematical Statistics with Applications, Mendenhall & Scheaffer, Duxbury Press, 1973.

12. References

1. P.D Chalkley and R.J Chester, 'Interim Report on Environmental Program - Durability of Graphite/Epoxy Honeycomb Specimens with Representative Damage and Repairs', Aeronautical and Maritime Research Laboratory, ARL-MAT-TM-397. AR-005-504
2. E-mail communication to Roger Vodicka, Colin Grey, AMRL, 12th February 1999.
3. H.B. Dexter and D.J. Baker, 'Flight Service Environmental Effects On Composite Materials And Structures', Advanced Performance Materials, Volume 1 Number 1, 1994, pp51-85
4. G. Clark, D.S. Saunders, T.J van Blaricun and M. Richmond, "Moisture Absorption in Graphite Epoxy Laminates", Composites Science and Technology, Vol. 39, 1990, pp355-375
5. R.J Chester and A.A. Baker, 'Environmental Durability of F/A-18 Graphite/Epoxy Composite', Polymers and Polymer Composites, Vol. 4, No. 5, 1996, pp315-323
6. XLStatistics, Rodney Carr, School of Management and Information Systems, Faculty of Business and Law, Deakin University, Warrnambool, Victoria 3280, AUSTRALIA
7. Effect of Strain Rate on the Inter-Laminar Shear Strength of Carbon-Fibre-Reinforced Laminates, J. Harding & L. Dong, Composites Science and Technology, 9 Sep 1992, p.353
8. The effect of moisture on the shear properties of carbon fibre composites, OM K. Joshi, *Composites*, Vol14, No3, July 1983, p.19
9. Interlaminar Shear Strength Fibre Reinforced Epoxy Composite under the Influence of Environments, M. Zhang & S.E. Mason, *Advanced Composites Letters*, Vol 7, No 1, 1998, p.28

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Appendix A: ILSS Test Results

The full listing of ILSS results is available from DSTO-AMRL in electronic format due to the size of the data set.

Specimen labelling and identification:

Each specimen was identified with a paint marker pen using a code, which indicated the test conditions for the particular specimen.

Wet tests: a1,a2, a3, = Tested at room temperature immediately on arrival

Wet tests hot: b1, b2, b3, = Tested at elevated temperature (100°C/steam) on arrival

Dry tests: c1, c2, c3, = Oven dried to constant weight at 80°C then tested at room temperature.

Dry tests hot: d1,d2, d3, = Oven dried at 80°C to constant weight then tested at 100°C.

Results Database:

The data base used to control the results was series of Excel work book spread sheets which recorded the following details (see Appendix A).

- Date of the test for each test condition.
- The number of days drying in the oven.
- The percentage weight loss.
- The cumulative weight loss.
- The thickness of each individual specimen.
- The width of each individual specimen.

The calculation for the apparent interlaminar shear strength (ILSS) was made using a mathematical formula preloaded into the spreadsheet along with the failure load and specimen dimensions.

Test equipment settings:

These settings are included for reference to chart paper copies of all test data accumulated on the INSTRON 1121 test machine.

Load cell rating	10 kN
Full scale chart deflection	5 kN
Chart speed	20 mm/min
Crosshead	0.5 mm/min
Record	Pen recorder

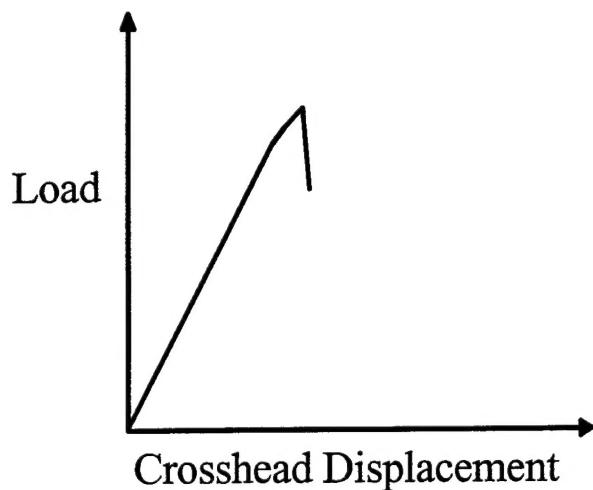
Sequence of ILSS test operations for hot tests:

- Remove chamber door
- Wait for steam to disperse
- Raise cross head
- Remove previous specimen
- Insert new specimen and check thermocouple position
- Lower crosshead close to specimen at a no load condition

- Close chamber door
- Wait for temperature to reach set-point
- When temperature recovered
- Start chart recorder and set correct crosshead speed
- Lower crosshead at test rate
- Check that all instrumentation running within prescribed limits(ie: temperature stability)
- Check that water level of steam generator is sufficient and check that load cell temperature is not excessive.

Typical ILSS Test Result:

The figure below shows a typical ILSS test result as captured on chart paper.



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R. Vodicka, J. van den Berg, R. Chester, B. Nelson

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DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION DOCUMENT CONTROL DATA		1. PRIVACY MARKING/CAVEAT (OF DOCUMENT)			
2. TITLE Long-Term Environmental Durability of F/A-18 Composite Material		3. SECURITY CLASSIFICATION (FOR UNCLASSIFIED REPORTS THAT ARE LIMITED RELEASE USE (L) NEXT TO DOCUMENT CLASSIFICATION) Document (U) Title (U) Abstract (U)			
4. AUTHOR(S) R. Vodicka, B. Nelson, J. van den Berg and R. Chester		5. CORPORATE AUTHOR Aeronautical and Maritime Research Laboratory PO Box 4331 Melbourne Vic 3001 Australia			
6a. DSTO NUMBER DSTO-TR-0826		6b. AR NUMBER AR-010-987		6c. TYPE OF REPORT Technical Report	7. DOCUMENT DATE June 1999
8. FILE NUMBER M1/9/650	9. TASK NUMBER AIR 98/201	10. TASK SPONSOR AIR	11. NO. OF PAGES 18	12. NO. OF REFERENCES 9	
13. DOWNGRADING/DELIMITING INSTRUCTIONS			14. RELEASE AUTHORITY Chief, Airframes and Engines Division		
15. SECONDARY RELEASE STATEMENT OF THIS DOCUMENT <i>Approved for public release</i>					
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No Limitations					
17. CASUAL ANNOUNCEMENT		Yes			
18. DEFTEST DESCRIPTORS Composite materials, Durability, Environmental tests, F/A-18 aircraft, Matrix materials, Epoxy composites , Degradation					
19. ABSTRACT The long-term environmental durability of AS4/3501-6 graphite/epoxy composite was evaluated by a ground exposure trial conducted at four locations within Australia and one in Malaysia for a period of between 468 and 520 weeks. The combined effects of moisture uptake, UV degradation, temperature, rainfall and wind on the strength of the samples were all evaluated. The coupons absorbed an average of about 0.9% moisture by weight over the length of the trial. The residual strength of the coupons was determined using the interlaminar shear strength (ILSS) test (ASTM D2344) both at ambient temperature and 100°C. A statistical analysis of the results determined that the combined effect of moisture and elevated temperature (100°C) decreases ILSS by about 10%. A small enhancement in ILSS at room temperature was noted for specimens which absorbed moisture over the period of the trial. This is likely to be due to the combined effects of stress relief and plasticisation of the matrix. No other significant degradation mechanism was identified in this study apart from a slight decrease in ILSS of about 5% for specimens which were coated with the standard F/A-18 paint scheme. This may be attributed to the solvents used in the application of the paint. However, the benefits of the paint protecting the composite surface from erosion and UV degradation far outweigh this structurally insignificant change in ILSS.					